PROJECT STATUS REPORT

September 14, 1991
Report No. AFR-529012-4
Report Period: August 15 - September 14, 1991

CONTRACT TITLE AND NUMBER:

"Epitaxial Growth of Single Crystal Diamond On Silicon"
Contract No. N00014-91-C-0101 AFR No. 529012
SDI/Innovative Science and Technology
Office of Naval Research

CONTRACTOR NAME:

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CONTRACT PERIOD:

May 15, 1991 - November 14, 1991

SUMMARY OF RECENT PROGRESS

Task 1 - Prepare Silicon Substrates

The specially polished Si(100) wafers have arrived. Each set is polished off-axis towards [110] at one degree intervals from 0-6°. Depositions on these wafers will begin in the near future. The deposition methods will include the torch, hot filament, and laser deposition reactors.

Task 2 - Prepare Buffer Layer on Si Substrate

We have performed a series of depositions on Si(100) surfaces using the hot filament reactor. The depositions are fairly short in duration (75 minutes) so that the nucleation is observable. The deposition conditions are total pressure = 10 torr, T_{sub} = 1000 °C, $T_{filament}$ ~ 1850 °C, and 1% CH_4 in H_2. The surfaces are prepared using various combinations of scratching, degreasing, and spin etching. The scratching preparation uses #6 diamond paste.

As shown in Figure 1, unscratched Si(100) has virtually no nucleation after 75 minutes of deposition. This is not surprising since other researchers find that 10-17 hours of CVD is required to cover fully the SiC buffer layer on unscratched Si (%%116(Belton et al., 1989)%%). Future work will extend deposition times to 10 hours in the hot filament reactor and shorter times in the high growth torch reactor.

By comparison, scratched Si(100) shows significant nucleation after 75 minutes of deposition (Figure 2). Figure 2a shows a view of the scratched silicon after ultrasonically washing in methanol but before deposition. Previous research has shown that residual silicon particles on a scratched silicon surface can serve as nucleation centers. No foreign particles are observed at this magnification (15,000x) indicating that residual silicon particles are removed by washing. After 75 minutes of deposition, however, nucleation occurs along the scratches and also in between scratches. The surface preparations are ultrasonic wash in methanol (Figure 2b), degreasing (Figure 2c), and degreasing and spin etching (Figure 2d). No matter which preparation is employed, the diamond nuclei are about 1μm in diameter, and there is no observable effect on nucleation density. There certainly is no evidence of single crystal epitaxy. Since the spin etching procedure is extremely clean and removes virtually all residual C and SiO_2 from the surface (%%117(Fenner et al., 1989)%%), one must conclude that scratching is mostly a physical effect rather than a chemical one. Another interesting result that jibes well with this conclusion is the high nucleation density of diamond on the edges of the wafer (Figure 3). Since the edge has many surface orientations off-axis to the (100) surface, one may hypothesize that the off-axis surfaces may behave similarly. Experiments in the next few months will confirm this hypothesis.

The literature searches for the various physical properties of silicon, diamond, and β-SiC is ongoing. Our interest focuses on surface energy, surface diffusion, and bulk diffusion of these materials. Results should be available within the next month.

Task 3 - Deposit Thick Diamond Films

This task has not yet begun.

Task 4 - Analysis of Films

This task is awaiting films produced under Task 2 and 3.

FUTURE WORK (through November 14)

- Deposit diamond films on spin etched Si(100) using the torch reactor. Explore the effects of scratching and deposition conditions.
- Continue preparations of SiC buffer layers on various on-axis and off-axis Si wafers using the hot filament reactor (spin etched at 0, 1, 2, 3, 4, 5, and 6°).
- Perform literature searches for the surface energy, surface diffusion, and bulk diffusion of silicon, diamond, and β-SiC.
- Attempt to grow the SiC buffer layer with a minimum of H atom flux to avoid roughening the Si surface.
- Perform diamond CVD on a single crystal of yttria stabilized zirconia because it has similar lattice constant to silicon and should not form a SiC layer.
- Deposit thicker diamond layers on the buffered wafers using the hot filament and torch reactors.
- Perform Raman, SEM, attenuated total reflectance (ATR), and X-ray analyses on deposited films.
- Prepare SiC buffer layers on various on-axis and off-axis Si wafers using pulsed laser deposition (PLD) of SiC and graphite.
- If necessary, prepared c-BN buffer layers on various on-axis and off-axis Si wafers using PLD.
- Repeat above experiments as needed.
- Prepare final report.

REFERENCES

1 D. N. Belton, S. J. Harris, S. J. Schmieg, A. M. Weiner, and T. A. Perry, Appl. Phys. Lett. **54**, 416 (1989).

2 D. B. Fenner, D. K. Biegelsen, and R. D. Bringans, J. Appl. Phys. **66**, 419 (1989).

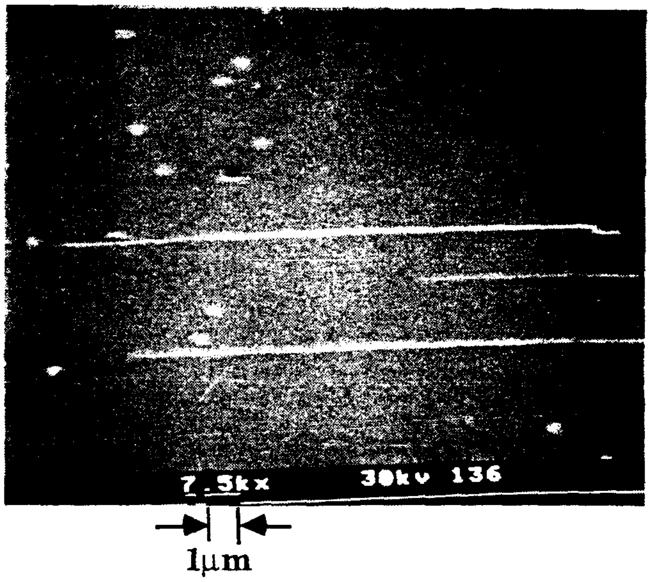

Figure 1 - Deposition on an Unscratched Si(100) Surface After Degreasing and Spin Etching.

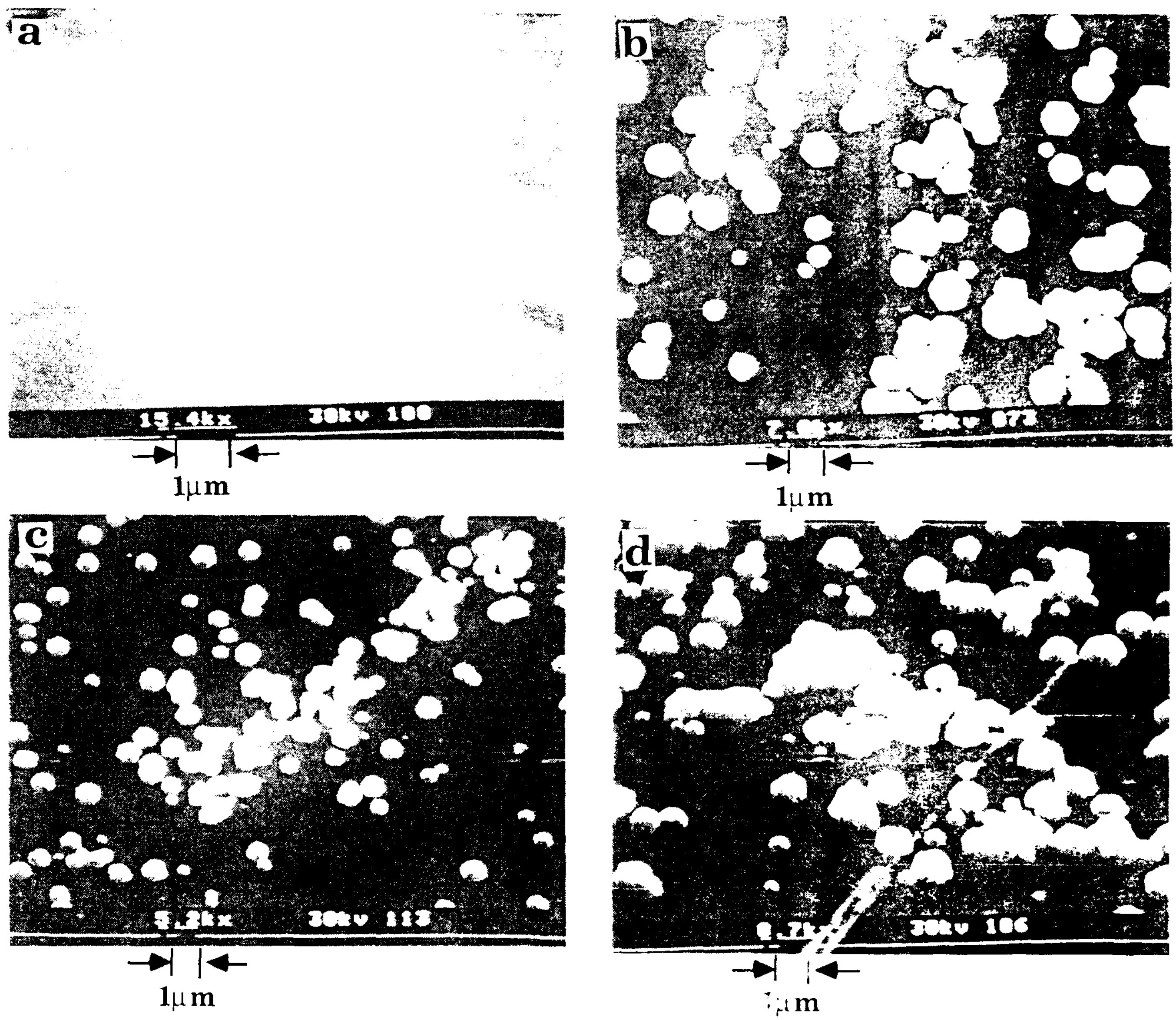

Figure 2 - Deposition on a Scratched Si(100) Surface. (a) After Washing in CH_3OH But Before Deposition; (b) After Washing Ultrasonically in CH_3OH; (c) After Degreasing; (d) After Degreasing and Spin Etching.

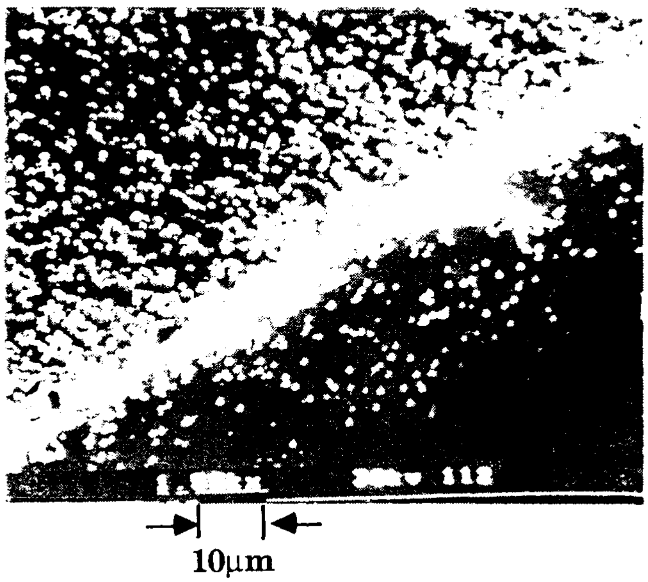

Figure 3 - Nucleation on the Edge of a Si(100) Wafer.